

09/923,629

Page 1

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L1 ANSWER 1 OF 4 CAPLUS COPYRIGHT 2003 ACS  
 ACCESSION NUMBER: 2002:119296 CAPLUS  
 DOCUMENT NUMBER: 136:167561  
 TITLE: Process for the isolation of sterols from the residues of fatty-acid or methyl-ester production  
 INVENTOR(S): Schwarzer, Joerg; Gutsche, Bernhard; Wollmann, Gerhard  
 PATENT ASSIGNEE(S): Cognis Deutschland GmbH, Germany  
 SOURCE: Eur. Pat. Appl., 7 pp.  
 CODEN: EPXXDW  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

| PATENT NO.  | KIND   | DATE     | APPLICATION NO.  | DATE     |
|---|--|----------|------------------|----------|
| EP 1179536  | A2   | 20020213 | EP 2001-118218   | 20010728 |
| R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO |  |          |                  |          |
| DE 10038442   | A1   | 20020221 | DE 2000-10038442 | 20000807 |
| US 2002058827   | A1   | 20020516 | US 2001-923629   | 20010807 |
| PRIORITY APPLN. INFO.: DE 2000-10038442 A 20000807  |  |          |                  |          |
| AB  | A process for obtaining sterols from the residue of fatty acid and/or Me ester prodn. is characterized by: (a) in the residue on hand free fatty acids are esterified with a polyhydroxy or lower monohydroxy alc., after that (b) the mixt. contg. partial glycerides is alcoholized at 90 - 145.degree. and a pressure of 2 - 10 bar over 2 - 20 mins with a lower alc. in the presence of a basic catalyst, (c) after the alcoholysis the excess lower alc. is distilled from the reaction mixt., (d) the alcoholysis catalyst as well as the included glycerin if necessary are sepd., (e) the fatty acid ester is distd. from the mixt. and (f) the bottoms contg. sterol and remaining partial glycerides through a further alcoholysis at 115 - 145.degree. and a pressure of 2 - 10 bars over 4 - 8 h leads to free sterol esters and fatty acid esters. Thus, the distn. residue from the cleavage of soybean oil is treated with glycerin in the presence of tin isooctanoate at 215.degree. and 7 mbar; the residue is then treated with MeOH contg. NaOMe at 137.degree. and 6 bar for 8 mins.; the Me esters are then distd. out; then residue is again treated with MeOH contg. NaOMe for 8 h at 120.degree. and 5 bar; the methanol is then flash evapd. and the catalyst neutralized with citric acid; the product mixt. is washed with H2O to give a product contg. 7.5% free sterols and 0.04% bound sterols; the sterol mixt. contains: 1.2% cholesterol, 1.8% brassicasterol, 23.1% campesterol, 15.3% stigmasterol, 48.9% .beta.-sitosterol, 2.2% .DELTA.5-avenasterol, 0.3% .DELTA.7-avenasterol and 0.05% citrostadienol. |          |                  |          |
| TI  | Process for the isolation of sterols from the residues of fatty-acid or methyl-ester production  |          |                  |          |
| AB  | A process for obtaining sterols from the residue of fatty acid and/or Me ester prodn. is characterized by: (a) in the residue on hand free fatty acids are esterified with a polyhydroxy or lower monohydroxy alc., after that (b) the mixt. contg. partial glycerides is alcoholized at 90 - 145.degree. and a pressure of 2 - 10 bar over 2 - 20 mins with a lower alc. in the presence of a basic catalyst, (c) after the alcoholysis the excess lower alc. is distilled from the reaction mixt., (d) the alcoholysis catalyst as well as the included glycerin if necessary are sepd., (e) the fatty acid  |          |                  |          |

L1 ANSWER 1 OF 4 CAPLUS COPYRIGHT 2003 ACS (Continued)  
 .DELTA.5-Avenasterol 23290-26-8P, .DELTA.7-Avenasterol  
 RL: PUR (Purification or recovery); SPN (Synthetic preparation); PREP (Preparation)  
 (process for the isolation of sterols from the residues of fatty acid or Me ester prodn.)  
 IT 56-81-5, Glycerin, reactions 67-56-1, Methanol, reactions  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (process for the isolation of sterols from the residues of fatty acid or Me ester prodn.)

L1 ANSWER 1 OF 4 CAPLUS COPYRIGHT 2003 ACS (Continued)  
 ester is distd. from the mixt. and (f) the bottoms contg. sterol and remaining partial glycerides through a further alcoholysis at 115 - 145.degree. and a pressure of 2 - 10 bars over 4 - 8 h leads to free sterol esters and fatty acid esters. Thus, the distn. residue from the cleavage of soybean oil is treated with glycerin in the presence of tin isooctanoate at 215.degree. and 7 mbar; the residue is then treated with MeOH contg. NaOMe at 137.degree. and 6 bar for 8 mins.; the Me esters are then distd. out; then residue is again treated with MeOH contg. NaOMe for 8 h at 120.degree. and 5 bar; the methanol is then flash evapd. and the catalyst neutralized with citric acid; the product mixt. is washed with H2O to give a product contg. 7.5% free sterols and 0.04% bound sterols; the sterol mixt. contains: 1.2% cholesterol, 1.8% brassicasterol, 23.1% campesterol, 15.3% stigmasterol, 48.9% .beta.-sitosterol, 2.2% .DELTA.5-avenasterol, 0.3% .DELTA.7-avenasterol and 0.05% citrostadienol.  
 ST sterol isolation prodn fatty acid methyl ester; glyceride prepn alcoholysis  
 IT Fatty acids, preparation  
 RL: PUR (Purification or recovery); SPN (Synthetic preparation); PREP (Preparation)  
 (esters); process for the isolation of sterols from the residues of fatty acid or Me ester prodn.)  
 IT Alcoholysis  
 Alcoholysis catalysts  
 Crystallization  
 Distillation  
 (process for the isolation of sterols from the residues of fatty acid or Me ester prodn.)  
 IT Fatty acids, preparation  
 Sterols  
 RL: PUR (Purification or recovery); SPN (Synthetic preparation); PREP (Preparation)  
 (process for the isolation of sterols from the residues of fatty acid or Me ester prodn.)  
 IT Alcohols, reactions  
 Coconut oil  
 Palm kernel oil  
 Palm oil  
 Rape oil  
 Soybean oil  
 Sunflower oil  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (process for the isolation of sterols from the residues of fatty acid or Me ester prodn.)  
 IT Glycerides, preparation  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (process for the isolation of sterols from the residues of fatty acid or Me ester prodn.)  
 IT 124-41-4, Sodium methoxide 30323-21-8, Tin isooctanoate  
 RL: CAT (Catalyst use); USES (Uses)  
 (process for the isolation of sterols from the residues of fatty acid or Me ester prodn.)  
 IT 57-88-5P, Cholesterol, preparation 83-46-5P, .beta.-sitosterol 83-48-7P, Stigmasterol 474-40-8P, Citrostadienol 474-62-4P, Campesterol 474-67-9P, Brassicasterol 18472-36-1P,

L1 ANSWER 2 OF 4 CAPLUS COPYRIGHT 2003 ACS  
 ACCESSION NUMBER: 2002:119296 CAPLUS  
 DOCUMENT NUMBER: 136:167560  
 TITLE: Process for the isolation of sterols from the residue of fatty acid ester production  
 INVENTOR(S): Gutsche, Bernhard; Bonakdar, Mehdi; Wollmann, Gerhard; Schwarzer, Joerg  
 PATENT ASSIGNEE(S): Cognis Deutschland GmbH, Germany  
 SOURCE: Eur. Pat. Appl., 8 pp.  
 CODEN: EPXXDW  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

| PATENT NO.  | KIND  | DATE     | APPLICATION NO.  | DATE     |
|---|---|----------|------------------|----------|
| EP 1179535  | A1  | 20020213 | EP 2001-118217   | 20010728 |
| R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO |   |          |                  |          |
| DE 10038456   | A1  | 20020221 | DE 2000-10038456 | 20000807 |
| US 2002082434   | A1  | 20020627 | US 2001-923626   | 20010807 |
| PRIORITY APPLN. INFO.: DE 2000-10038456 A 20000807  |   |          |                  |          |
| AB  | A process for obtaining sterols from the residue after distn. of alcoholized oils is characterized by: (a) alcoholysis of the mixt. contg. partial glycerides at a temp. of 115 - 145.degree. and a pressure of 2 - 10 over 5 - 20 mins. with a lower alc. contg. a basic catalyst, (b) after alcoholysis the excess lower alc. is distd. from the reaction mixt., (c) the alcoholysis catalyst is sepd. from the remaining glyceride, (d) the fatty acid alkyl ester is distd. from the mixt. and (e) the bottoms contg. sterol ester and residual partial glyceride through a further alcoholysis at 90 - 145.degree. and a pressure of 2 - 10 bar over 4 - 8 h leads to free sterol and fatty acid ester. Thus, the residue from the distn. of palm kernel oil was treated with MeOH contg. NaOMe at 122.degree. and 5 bar; after 8 min. the catalyst is neutralized with aq. citric acid; the Me ester is distd. at 180.degree. and 3 mbar; the bottoms are treated with more NaOMe in MeOH at 120.degree. for 5 h. |          |                  |          |
| REFERENCE COUNT:  | 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT  |          |                  |          |
| TI  | Process for the isolation of sterols from the residue of fatty acid ester production  |          |                  |          |
| AB  | A process for obtaining sterols from the residue after distn. of alcoholized oils is characterized by: (a) alcoholysis of the mixt. contg. partial glycerides at a temp. of 115 - 145.degree. and a pressure of 2 - 10 over 5 - 20 mins. with a lower alc. contg. a basic catalyst, (b) after alcoholysis the excess lower alc. is distd. from the reaction mixt., (c) the alcoholysis catalyst is sepd. from the remaining glyceride, (d) the fatty acid alkyl ester is distd. from the mixt. and (e) the bottoms contg. sterol ester and residual partial glyceride through a further alcoholysis at 90 - 145.degree. and a pressure of 2 - 10 bar over 4 - 8 h leads to free sterol and fatty acid ester. Thus, the residue from the distn. of palm kernel oil was treated with MeOH contg. NaOMe at 122.degree. and 5 bar; after 8 min. the catalyst is neutralized with aq. citric acid; the Me ester is distd. at 180.degree. and 3 mbar; the bottoms are treated with more NaOMe in MeOH at 120.degree. for 5 h. |          |                  |          |
| ST  | sterol isolation recovery fatty acid ester prodn; palm kernel oil alcoholysis methanol methoxide catalyst; glyceride prepn alcoholysis methanol methoxide catalyst  |          |                  |          |
| IT  | Fatty acids, preparation  |          |                  |          |

L1 ANSWER 2 OF 4 CAPLUS COPYRIGHT 2003 ACS (Continued)  
 RL: PUR (Purification or recovery); SPN (Synthetic preparation); PREP (Preparation)  
 (esters; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT Catalysts  
 (for alcoholysis; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT Palm kernel oil  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (for fatty acid ester prodn.; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT Alcohols, reactions  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (lower, for alcoholysis; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT Distillation  
 (of fatty acid esters; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT Alcoholysis  
 (of glycerides; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT Crystallization  
 (of **sterols**; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT Glycerides, preparation  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (partial alcoholysis of; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT **Sterols**  
 RL: PUR (Purification or recovery); SPN (Synthetic preparation); PREP (Preparation)  
 (process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT Fats and glyceridic oils, reactions  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (vegetable, alcoholysis of; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT 67-56-1, Methanol, reactions  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (alcoholysis by; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT 124-41-4, Sodium methoxide  
 RL: CAT (Catalyst use); USES (Uses)  
 (alcoholysis catalyst; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

L1 ANSWER 3 OF 4 CAPLUS COPYRIGHT 2003 ACS (Continued)  
**sterol** derivs., the free fatty acids present in the mixt. are esterified with an alc. The mixt. is then transesterified with an alc. in the presence of a basic catalyst.  
 After the transesterification, the excess lower alc. is distd. off from the reaction mixt. The transesterification catalyst and the glycerol present, if any, are removed and the fatty acid alkyl ester is distd. off from the mixt. Distn. of fatty acid alkyl esters can be accomplished with a packed column in sequence with a wiped film evaporator. The simultaneous recovery of tocopherol and **sterol** is possible. Tocopherols and **sterols** can be sepd. by the crystn. of **sterols** from a blend of org. solvents.

IT Alcohols, reactions  
 RL: PEP (Physical, engineering or chemical process); RCT (Reactant); PROC (Process); RACT (Reactant or reagent)  
 (recovery of tocopherols)

IT 64-18-6, Formic acid, uses 64-19-7, Acetic acid, uses 67-56-1, Methanol, uses 67-63-0, Isopropanol, uses 67-64-1, Acetone, uses 75-05-8, Acetonitrile, uses 78-93-3, MEK, uses 100-51-6, Benzyl alcohol, uses 108-87-2, Methylcyclohexane 108-88-3, Toluene, uses 109-94-4, Ethyl formate 109-99-9, THF, uses 110-54-3, Hexane, uses 110-82-7, Cyclohexane, uses 111-65-9, Octane, uses 141-78-6, Ethyl acetate, uses 142-82-5, Heptane, uses 1300-21-6, Dichloroethane 25265-68-3, Methyltetrahydrofuran 29222-48-8, Trimethylpentane  
 RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)  
 (recovery of tocopherols)

L1 ANSWER 3 OF 4 CAPLUS COPYRIGHT 2003 ACS  
 ACCESSION NUMBER: 1995:498453 CAPLUS  
 DOCUMENT NUMBER: 122:248289  
 TITLE: Recovery of tocopherols  
 Inventor: Hunt, Tracy K.; Jeromin, Lutz; Johannsbauer, Wilhelm; Gutsche, Bernhard; Jordan, Volkmar; Wogatzki, Herbert  
 PATENT ASSIGNEE(S): Henkel Corp., USA  
 SOURCE: PCT Int. Appl., 48 pp.  
 CODEN: PIXXD2  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

| PATENT NO.   | KIND | DATE     | APPLICATION NO. | DATE     |
|--|------|----------|-----------------|----------|
| WO 9504731   | A1   | 19950216 | WO 1994-US8481  | 19940801 |
| W: BR, CA, CN, JP, RU, UA  |      |          |                 |          |
| RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE |      |          |                 |          |
| CA 2168856   | AA   | 19950216 | CA 1994-2168856 | 19940801 |
| EP 712399  | A1   | 19950522 | EP 1994-924502  | 19940801 |
| EP 712399  | B1   | 20011114 |                 |          |
| R: AT, BE, CH, DE, ES, FR, GB, GR, IE, IT, LI, NL, PT, SE          |      |          |                 |          |
| BR 9407179   | A    | 19960917 | BR 1994-7179    | 19940801 |
| JP 09502701  | T2   | 19970318 | JP 1994-506442  | 19940801 |
| EP 992499  | A2   | 20000412 | EP 1999-118354  | 19940801 |
| EP 992499  | A3   | 20021211 |                 |          |
| R: AT, BE, CH, DE, ES, FR, GB, GR, IT, LI, NL, SE, PT, IE          |      |          |                 |          |
| EP 992500  | A2   | 20000412 | EP 1999-118355  | 19940801 |
| EP 992500  | A3   | 20000426 |                 |          |
| EP 992500  | B1   | 20020213 |                 |          |
| R: AT, BE, CH, DE, ES, FR, GB, GR, IT, LI, NL, SE, PT, IE          |      |          |                 |          |
| AT 208769  | E    | 20011115 | AT 1994-924502  | 19940801 |
| AT 213239  | E    | 20020215 | AT 1999-118355  | 19940801 |
| US 5616735   | A    | 19970401 | US 1995-531366  | 19950920 |
| US 5646311   | A    | 19970708 | US 1996-654483  | 19960528 |
| US 5670669   | A    | 19970923 | US 1996-654441  | 19960528 |

PRIORITY APPLN. INFO.:  
 US 1993-103628 A 19930806  
 US 1994-180592 A 19940113  
 EP 1994-924502 A3 19940801  
 WO 1994-US8481 W 19940801  
 US 1995-531366 A3 19950920

AB Starting from a mixt. contg. tocopherol, fats and/or fat derivs., more particularly fatty acids, and optionally **sterol** and/or **sterol** derivs., the free fatty acids present in the mixt. are esterified with an alc. The mixt. is then transesterified with an alc. in the presence of a basic catalyst.  
 After the transesterification, the excess lower alc. is distd. off from the reaction mixt. The transesterification catalyst and the glycerol present, if any, are removed and the fatty acid alkyl ester is distd. off from the mixt. Distn. of fatty acid alkyl esters can be accomplished with a packed column in sequence with a wiped film evaporator. The simultaneous recovery of tocopherol and **sterol** is possible. Tocopherols and **sterols** can be sepd. by the crystn. of **sterols** from a blend of org. solvents.

AB Starting from a mixt. contg. tocopherol, fats and/or fat derivs., more particularly fatty acids, and optionally **sterol** and/or

L1 ANSWER 4 OF 4 CAPLUS COPYRIGHT 2003 ACS  
 ACCESSION NUMBER: 1994:280367 CAPLUS  
 DOCUMENT NUMBER: 120:280367  
 TITLE: Separation of tocopherol and **sterols** from mixts. with fats and/or fatty acids.  
 Inventor: Jeromin, Lutz; Johannsbauer, Wilhelm; Gutsche, Bernhard; Jordan, Volkmar; Wogatzki, Herbert  
 PATENT ASSIGNEE(S): Henkel K.-G.A., Germany  
 SOURCE: Ger. Offen., 4 pp.  
 CODEN: GWXBX  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

| PATENT NO.   | KIND | DATE     | APPLICATION NO. | DATE     |
|--|------|----------|-----------------|----------|
| DE 4228476   | A1   | 19940303 | DE 1992-4228476 | 19920827 |
| DE 4228476   | C2   | 20020502 |                 |          |
| WO 9405650   | A1   | 19940317 | WO 1993-EP2207  | 19930818 |
| W: BR, CA, JP, US  |      |          |                 |          |
| RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE |      |          |                 |          |
| EP 656894  | A1   | 19950614 | EP 1993-919091  | 19930818 |
| EP 656894  | B1   | 19980225 |                 |          |
| EP 656894  | B2   | 20020612 |                 |          |
| R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL, SE                  |      |          |                 |          |
| JP 08500598  | T2   | 19960123 | JP 1993-506799  | 19930818 |
| AT 163416  | E    | 19980315 | AT 1993-919091  | 19930818 |
| ES 2112427   | T3   | 19980401 | ES 1993-919091  | 19930818 |
| BR 9306967   | A    | 19990112 | BR 1993-6967    | 19930818 |
| US 5627289   | A    | 19970506 | US 1995-387933  | 19950227 |

PRIORITY APPLN. INFO.:  
 DE 1992-4228476 A 19920827  
 WO 1993-EP2207 W 19930818

AB Title mixts., such as soybean oil steam distillate or tall oil, are treated with a lower alc., preferably MeOH, for esterification of free fatty acids, followed by transesterification, using a basic catalyst. The excess lower alc. is distd. off, and the catalyst and glycerol are removed by washing. After removal of the fatty acid alkyl esters by distn., the tocopherols and **sterols** are isolated by known methods. Esterification of the free fatty acids in the 1st stage is preferably carried out in the presence of strongly-acid ion exchangers.

TI Separation of tocopherol and **sterols** from mixts. with fats and/or fatty acids.

AB Title mixts., such as soybean oil steam distillate or tall oil, are treated with a lower alc., preferably MeOH, for esterification of free fatty acids, followed by transesterification, using a basic catalyst. The excess lower alc. is distd. off, and the catalyst and glycerol are removed by washing. After removal of the fatty acid alkyl esters by distn., the tocopherols and **sterols** are isolated by known methods. Esterification of the free fatty acids in the 1st stage is preferably carried out in the presence of strongly-acid ion exchangers.

ST tocopherol **sterol** sepn fat fatty acid; soybean oil distillate

IT Soybean oil  
 RL: BIOL (Biological study)  
 (steam distillate of, tocopherols and **sterols** sepn. from)

IT Tall oil  
 RL: BIOL (Biological study)  
 (tocopherols and **sterols** sepn. from)

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L1 ANSWER 4 OF 4 CAPLUS COPYRIGHT 2003 ACS (Continued)

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L1           4 S STEROL? AND BASIC CATALYST AND ALCOHOL